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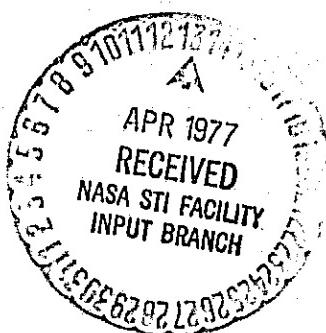
FRICTION AND WEAR BEHAVIOR OF GRAPHITE
FIBER REINFORCED POLYIMIDE COMPOSITES

by Robert L. Fusaro and Harold E. Sliney

Lewis Research Center

Cleveland, Ohio 44135

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REINFORCED POLYIMIDE COMPOSITES

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National Aeronautics and Space Administration

Lewis Research Center

Cleveland, Ohio 44135

ABSTRACT

The friction and wear rate characteristics of 50/50 (weight percent) graphite fiber-polyimide composites were studied by sliding metallic hemispherically tipped riders against disks made from the composites. Two different polyimides and two different graphite fibers were evaluated. Also studied were such variables as the effect of adding 10 percent weight additions of powdered $(CF_{1.1})_n$, CdI_2 , or CdO ; the effect of moisture in an air atmosphere; the effect of temperature; and the effect of different sliding speeds. In general, wear to the metallic riders was negligible and composite wear increased at a constant rate as a function of number of sliding cycles.

INTRODUCTION

The use of self-lubricating materials in dry bearings, dynamic seals, and gears is continually increasing (1-5). Materials are needed which will give low friction, low wear, high load capacity and are able to withstand high temperatures. Because of this need, considerable research has been conducted on reinforced polymers (5-17).

Reinforcing fibers have been used to improve the mechanical properties (such as strength and stiffness) of polymers for several years now. When graphite is used as the reinforcing fiber, the added advantage of self-lubrication can be achieved. In addition graphite fibers can improve the electrical and thermal conductivity of polymers, and the latter can be very important to friction. One new class of self-lubricating composites, graphite fiber reinforced polyimide (GFRPI), has been shown to have very good tribological properties up to $340^{\circ} C$ (7, 14, 15, 17). The friction, wear, and dynamic load capacity of one type of graphite fiber-reinforced polyimide composite has been determined by means of an oscillating spherical bearing tester (14-16). The results indicated that this composite material functions extremely well as a self-lubricating bearing material.

An objective of this study was to determine if improvements in performance of the composites mentioned above could be achieved by employing a different polyimide, a different type of graphite fiber, or by adding powdered solid lubricant additives to the composites. Besides trying to formulate an improved composite material, a further objective was to investigate the basic friction and wear properties of these composite materials. Experimental variables such as sliding speed, the absence of H_2O in the atmosphere, and high temperatures were studied.

Since spherical bearing tests are very time consuming, a bench test type of apparatus (pin-on-disk) was used to evaluate the composite materials. Thus another objective was to ascertain the extent to which a bench test type of apparatus could predict the friction and wear characteristics of composite materials. To accomplish this, the results of this study were compared to those from spherical bearing tests (16).

MATERIALS

The composite disks used in this study were made from a 50/50 (by weight) mixture of graphite fibers and polyimide resin. The fibers were chopped into lengths of 6.4×10^{-3} m (0.25 in.) and were randomly dispersed throughout the polyimide matrix. Two different types of graphite fibers were evaluated. The fiber designated as type "L" had a tensile strength and an elastic modulus which could be considered low. The fiber designated as type "H" had what might be called a medium tensile strength and a high elastic modulus. Table 1 gives typical properties for the two types of fibers used.

Two different types of polyimide resins were also evaluated. Both were formulated so that a low level of voids in the final cured polymer could be achieved. The polyimide designated type "A" was an addition-type polyimide and was highly crosslinked. Its molecular structure is given in Fig. 1. The second polyimide, designated as type "C" was a condensation type of polyimide which was linear, amorphous, and essentially noncrosslinked. Its molecular structure is shown in Fig. 2.

Three different combinations of the above fibers and polyimides were evaluated: the addition polyimide and the low modulus fiber (composite type "AL"), the condensation polyimide and the low modulus fiber (composite type "CL"), and the condensation polyimide and the high modulus fiber (composite type "CH").

Ten percent (by weight) of either graphite fluoride ($CF_{1.1}$)_n powder, cadmium iodide (CdI_2) powder, or cadmium oxide (CdO) powder was added to type "CH" composites. These solid lubricant additives were incorporated into the polyimide/graphite fiber mixture before polymerization.

The rider material was 440C stainless steel, which was hardened to Rockwell C-60. The riders were hemispherically tipped (0.476) and ground to a finish of 10^{-7} m (4 μ in.).

APPARATUS DESCRIPTION

A hemisphere-on-flat type of sliding friction apparatus was used in this study. The friction specimens (Fig. 3) consisted of a stationary (0.476 cm radius) hemispherically tipped metallic rider in sliding contact with a flat (6.3 cm diam) composite disk. The wear track diameter on the disk could be varied by changing the position of the rider; therefore several tests could be performed on each disk, on diameters that ranged from 3.8 to 5.8 cm. The apparatus was equipped with a variable-speed motor and gear reduction system so that the rotational speed could be controlled. The composite disk was heated by induction by means of a metallic susceptor located around the circumference of the disk. The temperature was monitored with an infrared pyrometer.

EXPERIMENTAL PROCEDURE

The rider and disk were inserted into the friction apparatus, and the test chamber was sealed. Moist air (10 000 ppm H_2O) or dry air (<20 ppm H_2O) was then purged through the chamber for 15 minutes. The flow rate was 1500 cubic cm per minute and the volume of the chamber was 2000 cubic cm. Two test temperatures were used in the study, 25° and 300° C. The 300° C temperature was obtained by slowly heating a susceptor located around the circumference of the disk by induction heating, and then holding it at temperature for 10 minutes to allow it to stabilize. The load used was 1 kg and the sliding speed was 2.5±0.5 m per second (1000 rpm on tracks that varied from 3.8 to 5.8 cm diam).

At various intervals during the experiments, the tests were stopped and the specimens examined. Wear was determined by taking surface profiles of the wear tracks on the graphite fiber-reinforced polyimide composite disks and determining the cross-sectional area of the material worn away. The riders were not removed from their

holders, and locating pins in the apparatus insured that the riders and disks were returned to their original positions.

RESULTS AND DISCUSSION

Friction and Wear in a Moist Air Atmosphere

The first series of experiments on the composites was conducted in what might be considered a typical atmospheric condition, moist air of approximately 50 percent relative humidity at 25° C (10 000 ppm H₂O). Wear of the three different composites is shown in Fig. 4 as a function of the number of sliding revolutions. The general trend of the wear is that it increases in a linear manner (from zero) as a function of the number of sliding revolutions. By fitting the best straight lines to the points, the wear rates were found to be 1.3×10^{-10} cm² per cycle for the type "AL" composite, 2.7×10^{-10} cm² per cycle for the type "CL" composite, and 5.1×10^{-10} cm² per cycle for the type "CH" composite. Similar tests were performed at 300° C and the following wear rates were determined: type "AL", 1.5×10^{-10} cm² per cycle; type "CL", 1.8×10^{-10} cm² per cycle; and type "CH", 2.2×10^{-10} cm² per cycle.

Figure 5 gives representative surface profiles of composite wear tracks after 300 000 cycles of sliding. The figure illustrates the differences in the wear that has taken place and indicates that at 25° C the type "A" polyimide is better than type "C" and that type "L" fibers are better than type "H" fibers; however at 300° C there is not a great deal of difference in wear.

Figure 6 gives representative friction traces for the tests conducted at 25° and 300° C. Basically the same relative ranking of the composites occurred when the friction coefficients were compared as when the wear rates were compared, except that the difference is not as great.

The large difference in friction coefficient between 25° and 300° C is quite interesting. At 300° C the composites seemed to exhibit some sort of "run-in" phenomena, whereas at 25° C they did not. In some previous work (18-20), which was done on a different polyimide, it was discovered that a friction and wear transition occurred as a function of temperature. In those studies it was found that a transition from high wear-high friction to low wear-low friction occurred for this polyimide (without any additives) in a dry argon or dry air (<20 ppm H₂O) atmosphere at 40°±10° C. When

moisture was present in the atmosphere the transition was shifted to an even higher temperature.

It may be that a similar transition also occurs in the polyimides which were used in this study. This would help to explain the "run-in" phenomena and the considerably lower friction coefficients obtained at 300° C than at 25° C. Whether or not this is the case should be the subject of a future study.

Bench test results have received criticism because they only approximate the actual application. For that reason the results of this study were compared to the results of Ref. 16, where the composite material was actually investigated in a potential end use, a self-aligning plain bearing. The comparison is given in Table 2, where the wear rate of type "AL" composites are given. Two wear rates are given for the bearings since two types of liners were used in that study.

In the bearing tests of Ref. 16, wear rates were expressed as wear volume per sliding distance per unit load. For comparison purposes those units have been converted to the ones used in this paper, cross-sectional area per cycle of sliding per unit load. Dimensionally the units are the same; however when the material being worn is not in continuous contact, it seems more appropriate to express wear as a function of repeated passes.

The closeness of the wear rates, as determined by the two methods, indicates that bench tests can be used to evaluate materials and to determine wear rates. The important consideration is to determine what variables are relevant and should be used in the comparison.

One should be very cautious in making comparisons, however, since wear is a very complicated phenomena. Wear of polymers, for example, can be affected by such experimental conditions as temperature, load, sliding speed, and the atmosphere in which the specimens are evaluated. Another important consideration, often overlooked, is the geometry of the sliding specimens. The geometry not only affects the contact stresses, it can also affect the direction of the shear stresses induced in the polymer and also the nature of transfer film formation. The latter two phenomena are areas in which further research is needed.

Powdered solid lubricant additives have often been used in composite materials to improve their friction and wear properties. Cadmium oxide (CdO) and cadmium iodide (CdI_2) are well known compounds which are added to graphite formulated solid lubricant films to improve their lubrication properties. Ten percent (by weight) of each of these compounds was added to type "CH" composites to determine if improvements could be achieved in graphite-fiber-reinforced polyimide composites. Ten percent (by weight) of the relatively new solid lubricant material, graphite fluoride ($\text{CF}_{1.1}$)_n, was also added to type "CH" composites and evaluated. Previous studies (21-24) indicated this material has excellent intrinsic lubricating properties.

The effect of solid lubricant additives on the friction coefficient of type "CH" composites is shown in Fig. 7. These composites were evaluated under the same conditions as the previous tests, that is, a load of 1 kg, a speed of 1000 rpm, and in a moist air atmosphere. The results indicate that CdI_2 and $\text{CF}_{1.1}$ both improved the friction characteristics of type "CH" composites; although at 300° C, for about 30 000 cycles, the friction coefficient of the composite with the CdI_2 additive rose to a value as high as 0.24. Additions of CdO did not improve the friction coefficient compared to the base composite and since wear was about 100 times greater than the base composite, further testing with this additive was discontinued.

The effect of additives on the wear of type "CH" composites after sliding for 300 000 cycles at 25° and 300° C is shown in Fig. 8. CdI_2 and $\text{CF}_{1.1}$ additives both improved the wear properties of the base composite at 25° C; however, at 300° C, negative results were obtained. These results are consistent with the reasoning that the graphite fibers are providing the lubrication at 25° C, and that these additives assist the graphite. The higher wear at 300° C may have simply been due to a weakening of the structure by the additives at this higher temperature. It is also noteworthy to point out that materials should not be evaluated on friction considerations alone, as Figs. 7 and 8 exemplify.

Friction and Wear in a Dry Air Atmosphere

It has been mentioned previously that the friction and wear properties of polymers can be affected by the atmosphere in which the polymer specimens slide. It is also well

known that graphite functions better as a lubricant under moist conditions. Since potential applications for these self-lubricating composites are in an absence or a minimum of H₂O in the atmosphere (such as in aircraft or the space shuttle), it was of interest to determine what the effect an absence of moisture would have on the lubricating properties of these composites.

Similar experiments to those conducted in moist air (10 000 ppm H₂O) were therefore also conducted in a dry air (20 ppm H₂O) atmosphere. The same load (1 kg) and rotational speed (1000 rpm) were used. Figure 9 gives the friction traces and Fig. 10 gives surface profiles for these experiments which were run for a total of 300 000 cycles. The results obtained in dry air were considerably different than those obtained in moist air. In moist air, lower friction coefficients were obtained at 300° C than at 25° C, but in dry air just the opposite occurred (with the exception of type "AL" composites). The same reversal in results occurred when wear rates of the base composites were compared. Figure 11 gives a summary of all the wear rate and average friction coefficient results conducted in this study.

Interpretation of the results is open to speculation. If graphite were the dominant factor in providing the lubrication, higher friction and also higher wear would be expected in the dry air atmosphere. Indeed this happened at 300° C; but not at 25° C. A consideration that might be made is that absorbed H₂O would be less of a factor at 300° C than at 25° C, since it would desorb faster at this higher temperature. However, this does not explain why lower friction and wear results were obtained in dry air than in moist air at 25° C. If graphite fibers were the dominant factor in the lubrication process of these composites, the best one would expect would be equivalent results in moist and dry air.

Considering all the results from this study, it appears that the friction and wear properties of these composites were controlled by the interaction of the polyimide and graphite fiber properties. The solid lubricant additives were found to give some benefit, but the overriding consideration appears to be the properties of the polyimide and graphite fiber.

The lubricating properties of graphite are fairly well known. Polyimide, however,

is a relatively new polymer; and the name polyimide is a generic term for thousands of different possibilities that might be synthesized, each of course possessing different properties. It is therefore very obvious that a more basic knowledge of the friction and wear characteristics of polyimide is necessary in order to be able to formulate the best possible graphite fiber reinforced polyimide composite.

Effect of Speed on Friction Coefficient

The effect of linear sliding speed on the friction coefficient of the various graphite fiber reinforced polyimide composites was also investigated in this study. The study was conducted in a moist air atmosphere (10 000 ppm H₂O), at 25° C, under a load of 1 kg, and at speeds of 0.5, 6, 60, 120, 240, 480, and 800 rpm. The track diameter was 5.2 cm, thus the linear sliding speeds were 0.14, 1.6, 16, 33, 65, 130, and 218 cm per second. Ten-minute tests at each sliding speed were conducted starting at the 6-rpm speed. The specimens were disengaged when speed was increased or decreased and the order of testing was to increase the speed to 800 rpm and then to decrease it to 0.5 rpm.

Figure 12 presents the results of this study. Except for the type "CH" composite with the CdO additive, the average friction coefficient did not greatly depend on sliding speed. The most noticeable effect of increased sliding speed was to average out the value of friction coefficient obtained. At slower speeds, due to the anisotropic nature of the composites, the friction coefficient varied markedly as the rider slid around the disk track. Nominally the fibers were randomly dispersed throughout the polyimide matrix, but such things as local variations in fiber density, fiber orientation, etc., apparently had an influence on the friction coefficients of these composites.

This influence was also seen in the wear rate experiments in the first part of the paper. Surface profiles of the wear track at various positions around the track showed that wear was not uniform. Table 3 gives percent variations of the cross-sectional area of the composite wear tracks. It appears that wear and friction are both influenced by such factors as fiber orientation and fiber density. The data of Table 3 give an indication of the reproducibility of wear rates obtained in this study. While all con-

ditions were not repeated, the ones that were fell within the limits of the percent variations of Table 3.

SUMMARY OF RESULTS

Friction and wear experiments conducted on composites made from graphite fibers and polyimide resin (50/50 weight percent) indicate the following.

1. Better results were obtained with the addition type of polyimide than with the condensation polyimide.
2. Better results were obtained with the low modulus-low strength type of graphite fibers than with the high modulus-medium strength type of graphite fibers.
3. Wear rates were found to be relatively constant for the duration of the tests, and the values obtained compared favorably to the wear rates determined previously in a self-aligning plain bearing tester.
4. Considering the effects of atmosphere, friction and wear of these composites appear to be controlled by the combined interaction of the polyimide and graphite fiber properties.
5. In moist air, 10 weight percent additions of either graphite fluoride or cadmium iodide gave improved friction and wear results at 25° C., but not at 300° C. In dry air, graphite fluoride gave improved results at both temperatures, but cadmium iodide gave negative results at both temperatures. Cadmium oxide additions were found to be detrimental at all test conditions evaluated.
6. Average friction coefficients did not greatly depend upon sliding speed in the range of 0.5 to 218 cm per second.

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TABLE 1. - TYPICAL GRAPHITE FIBER PROPERTIES

Property or characteristic	Type "L"		Type "H"	
	English units	SI units	English units	SI units
Tensile strength	9.0×10^4 lb/in ²	6.2×10^8 N/m ²	2.8×10^5 lb/in ²	2.0×10^9 N/m ²
Elastic modulus	5.0×10^6 lb/in ²	3×10^{10} N/m ²	5.7×10^7 lb/in ²	3.9×10^{11} N/m ²
Length	0.25 in.	6.4×10^{-3} m	0.25 in.	6.4×10^{-3} m
Diameter	3.3×10^{-4} in.	8.4×10^{-6} m	2.6×10^{-4} in.	6.6×10^{-6} m
Specific gravity	1.4	1.4	1.4	1.4

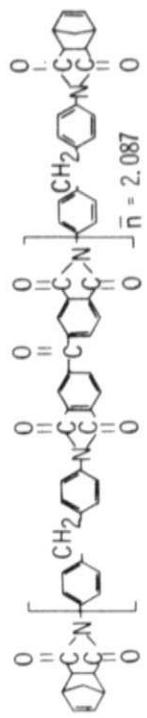
TABLE 2. - COMPARISON OF WEAR RATES
OBTAINED FROM DIFFERENT TEST APPARATUS
(TYPE "AL" POLYIMIDE COMPOSITES)

Temper- ature, °C	Wear rate (cm ² /cycle-kg×10 ⁻¹⁰)		
	Pin-on-disk	Self-aligning plain bearings ^a	
		Molded liner	Insert liner
25	1.3 ± 0.4	1.2 ± 0.4	2.0 ± 1.0
300	1.5 ± 0.3	1.2 ± 0.4	2.0 ± 1.0

^aData from Ref. 16.

TABLE 3. - MAXIMUM VARIATION OF THE CROSS-
SECTIONAL AREA OF THE COMPOSITE WEAR TRACKS

Composite type	Percent variation			
	Moist air (10 000 ppm H ₂ O)		Dry air (<20 ppm H ₂ O)	
	25° C	300° C	25° C	300° C
"AL"	34	19	17	46
"CL"	21	35	25	28
"CH"	30	24	32	23
"CH" and CF _{1,1}	33	20	25	17
"CH" and CdI ₂	63	48	36	61



(a) IMIDIZED PREPOLYMER.

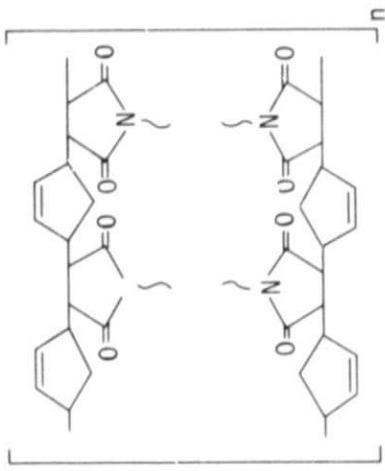


Figure 1. - Addition-type of polyimide polymer (Type A) used in this study.

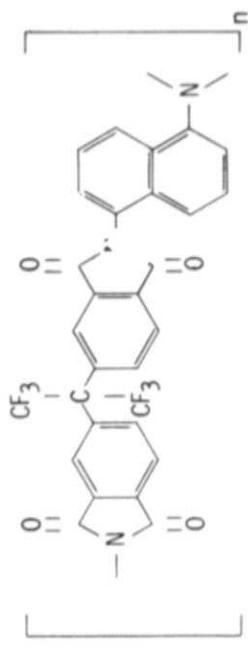


Figure 2. - Condensation-type of polyimide polymer (Type C) used in this study.

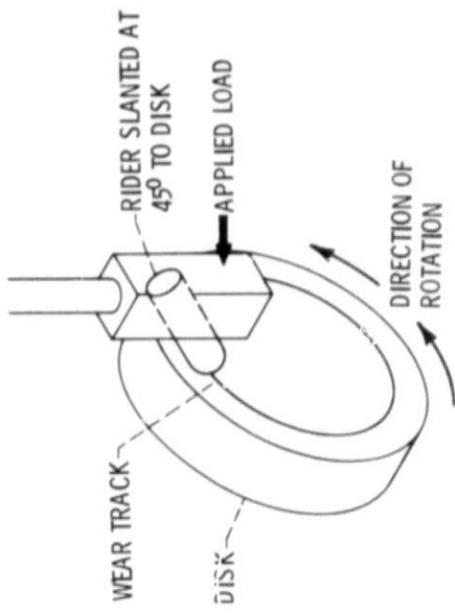


Figure 3. - Schematic diagram of friction specimens.

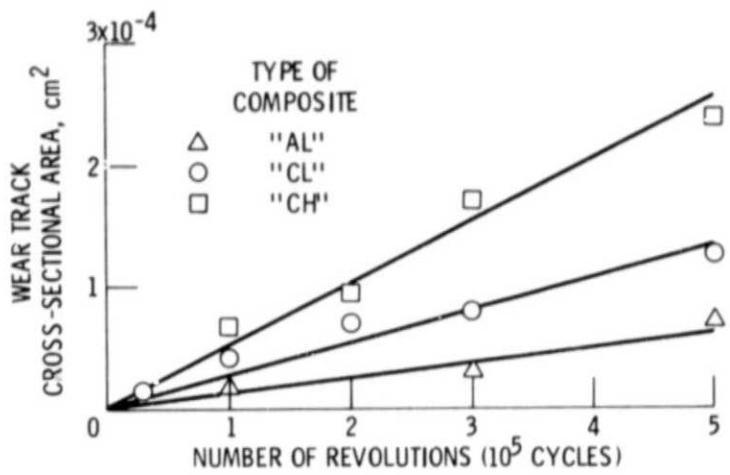


Figure 4. - Composite film wear at 25° C in a moist air atmosphere (10 000 ppm H₂O) as a function of the number of sliding revolutions.

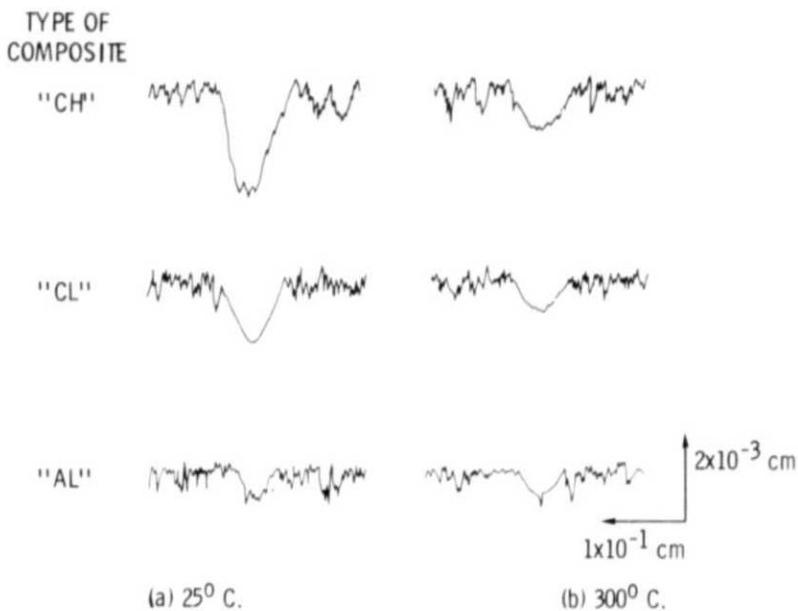


Figure 5. - Surface profiles of composite wear tracks taken after 300 000 cycles of sliding in a moist air atmosphere (10 000 ppm H₂O).

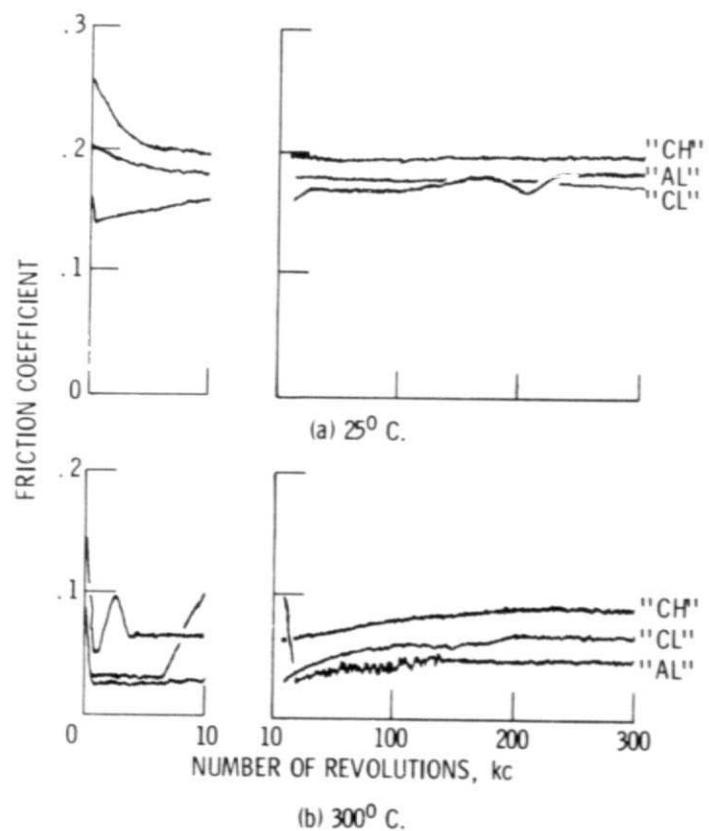


Figure 6. - Friction coefficient as a function of sliding revolutions in a moist air (10 000 ppm H₂O) atmosphere.

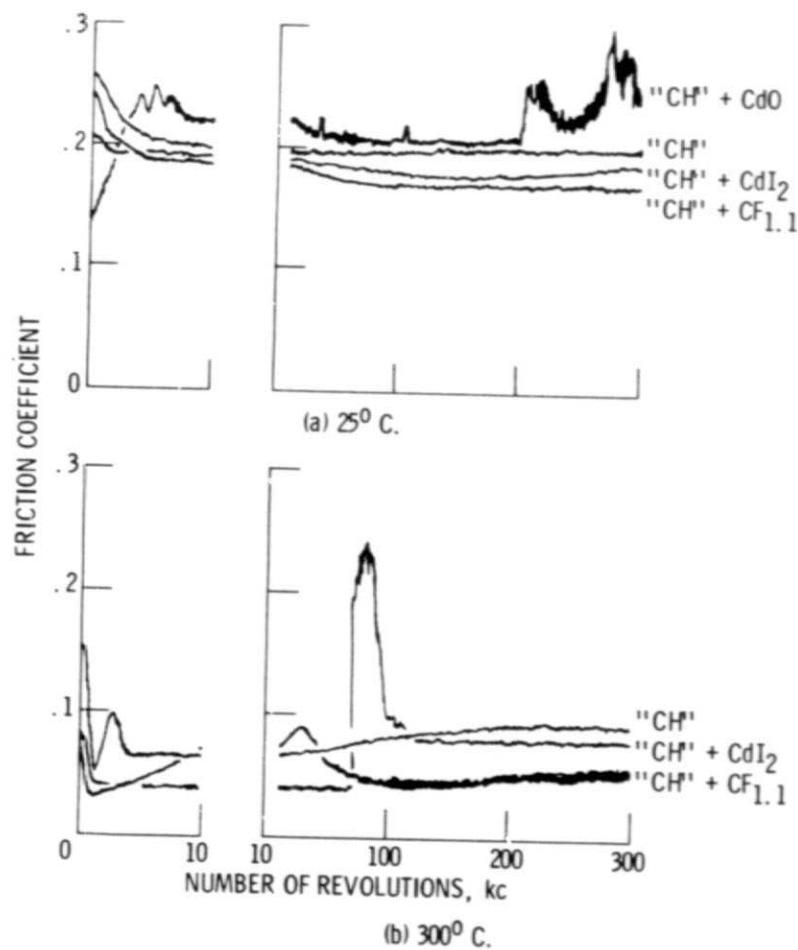


Figure 7. - Effect of solid lubricant additives on the friction coefficient of Type-'CH' polyimide composites.

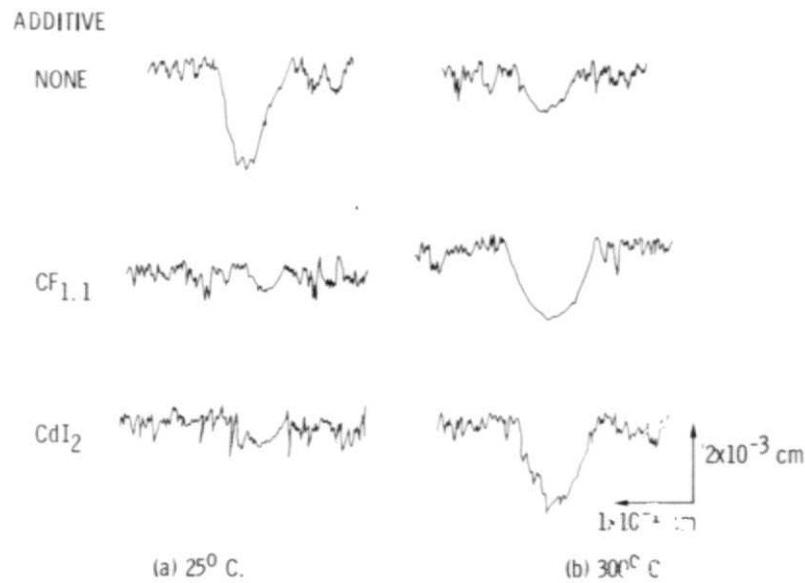


Figure 8. - Effect of additives on the wear of type "CH" composites after sliding in a moist air atmosphere (10 000 ppm H₂O) for 300 000 cycles.

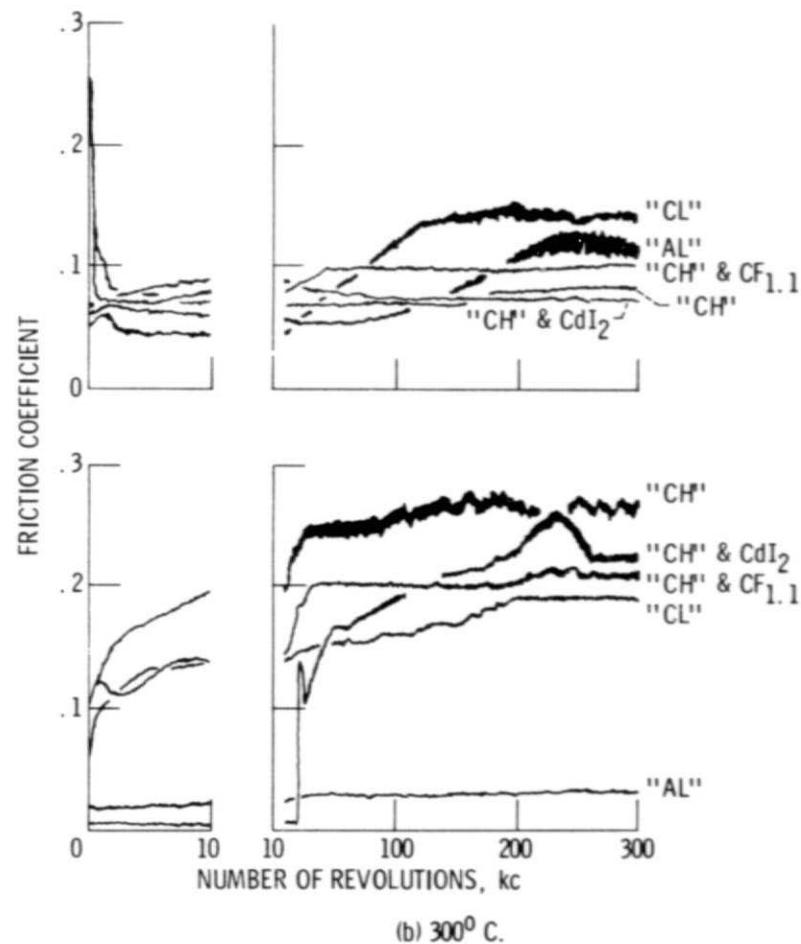


Figure 9. - Effect of a dry air atmosphere (<20 ppm H₂O) on the friction coefficient of graphite fiber reinforced polyimides with and without solid lubricant additives.

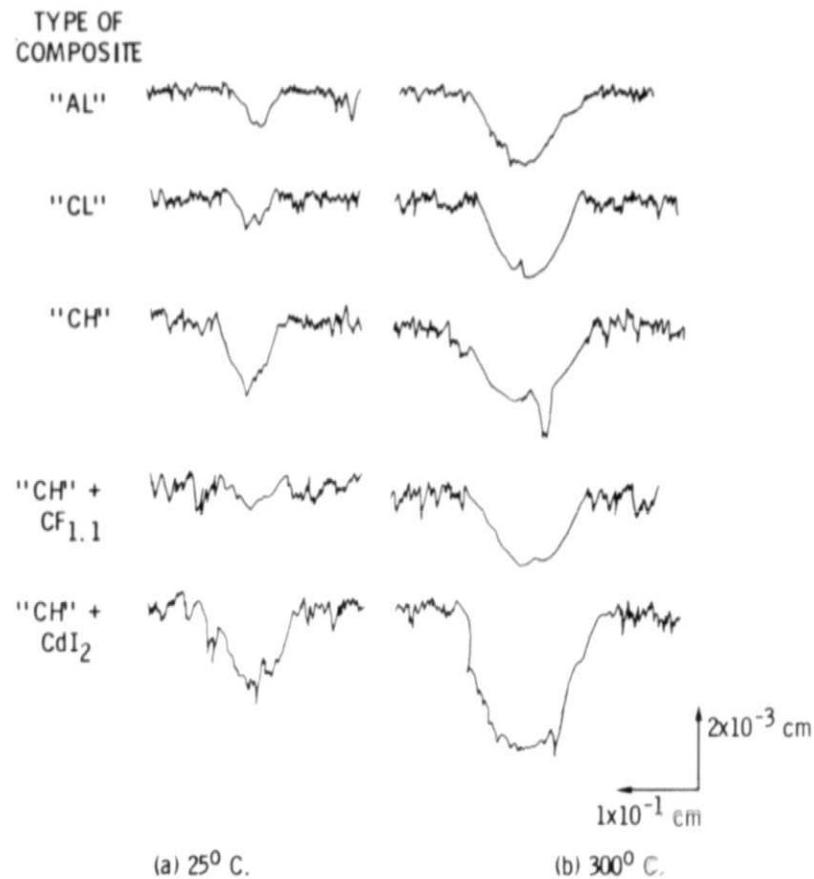


Figure 10. - Effect of a dry air atmosphere (<20 ppm H₂O) on the wear of polyimide composites after 300 000 cycles of sliding at (a) 25° C and (b) 300° C.

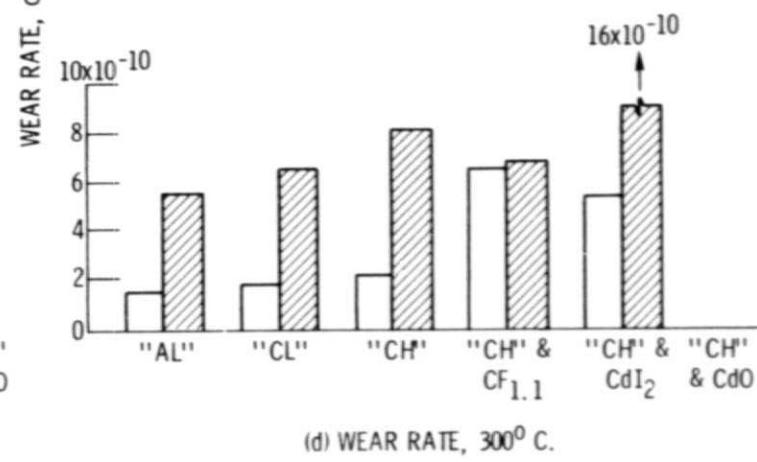
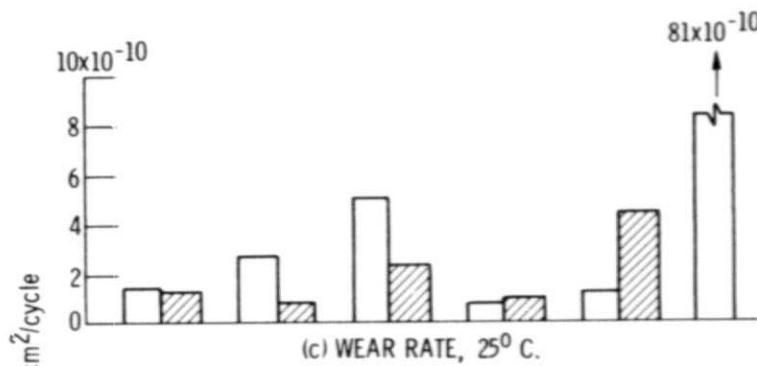
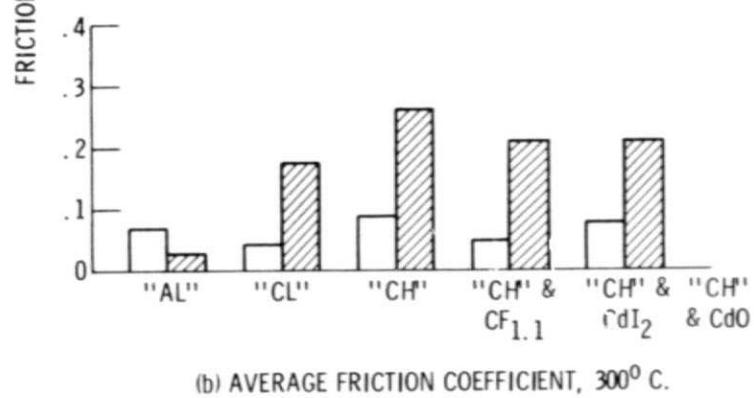
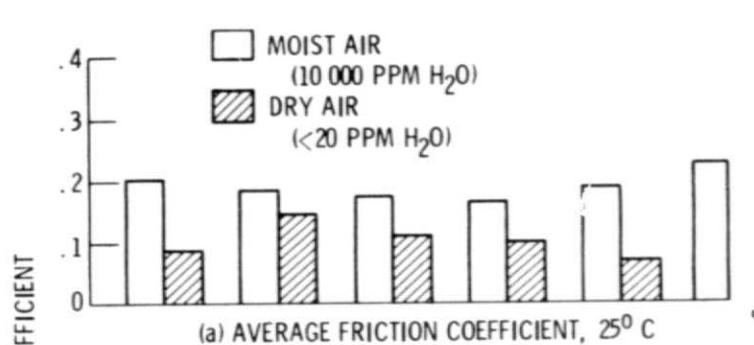


Figure 11. - Effect of atmosphere on the average friction coefficients at (a) 25° C and (b) 300° C and on the average wear rates at (c) 25° C and (d) 300° C.

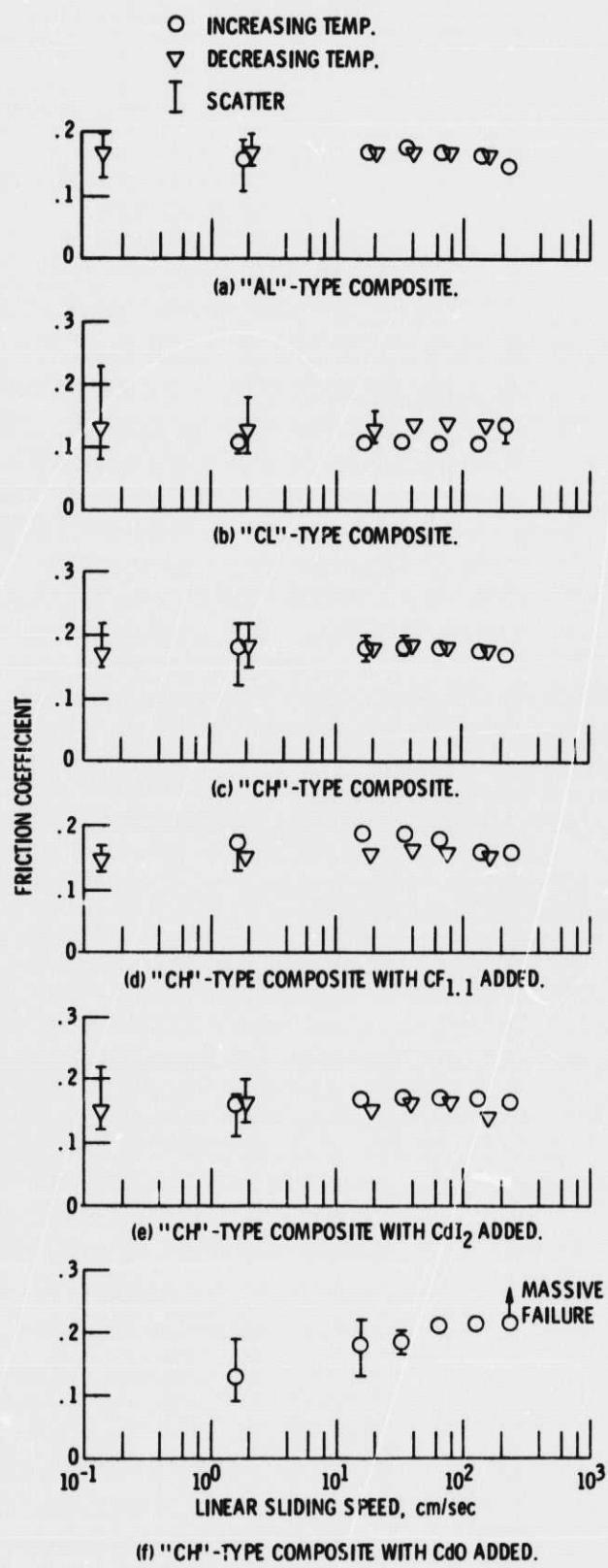


Figure 12. - Effect of sliding speed on the friction coefficient of six different polyimide composites which were evaluated in a moist air atmosphere (10 000 ppm H_2O).